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Atlas of Source Emission Particles

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ATLAS OF SOURCE EMISSION PARTICLES

by

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ENVIRONMENTAL SCIENCES RESEARCH LABORATORY
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PREFACE

A major effort in environmental protection is to develop measurement technology needed for source and ambient air monitoring of pollutant emissions. These efforts depend on detection, identification, and quantification of specific pollutants, and assessment of their effects. The Emissions Measurement and Characterization Division conducts studies to identify and determine the chemical and physical nature of both stationary and mobile source emissions.

One important means of making such measurements is electron microscopy. The application of this technology to the characterization of emissions from power generation, various manufacturing operations, mining and quarrying, and automotive commerce has led to the compilation of data that has been indispensible to developing instrumentation and methodology for the safe disposal of discarded consumer goods and industrial scrap and wastes.

ABSTRACT

An atlas of various source emission particles characterized by electron optical techniques has been compiled for use by air pollution investigators. The particles studied were emitted by mobile, stationary, and natural sources. Sources included automobiles, manufacturing operations, power plants, smelters, mining and quarring. Filter media and sample preparation methodology as well as morphological and chemical data are presented.

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SECTION 1

INTRODUCTION

The methodology and instrumentation for controlling various source emissions depends on the physical and chemical nature of these emissions. There are as many instruments designed to measure particle size as there are to determine the chemistry of particles. But to ascertain the morphology and chemistry of each particle there is only one suitable instrument, the electron microscope.

There are two types of electron microscopes -- the transmission and the scanning. Both instruments can be fitted with x-ray spectrometers for analytical purposes. The transmission electron microscope (TEM) can be used for particulate sizes down to 0.05 μm with good analytical results using energy-dispersive x-ray fluorescence (EDX); selected area electron diffraction (SAED) makes possible the compound identification in much the same manner as x-ray diffraction. However, identification by morphology alone is limited, and instrument geometry restricts the size of the sample to be examined.

The scanning electron microscope (SEM) while not limited to sample size, does not have the TEM's resolution. The SEM, fitted with EDX is capable of obtaining the chemical composition of the particles plus rendering excellent morphological data from secondary electrons emitted from the sample. Both types of scopes are indispensible in the study of particulate material because of the particular image-forming mechanism peculiar to each instrument.

Studies of large numbers of samples from many varied sources result in data that is suitable for presentation as an atlas. One which is in general use is called the "Particle Atlas" (1) and covers almost every conceivable material. Other publications of more limited scope are the "Asbestos Fiber Atlas" (2) and "Identification of Selected Silicate Minerals and their Asbestiform Varieties by Electron Optical and X-Ray Techniques" (3).

This atlas contains data from sources such as the manufacture of insulating materials, textile, smelting, steel, fertilizer, light weight composite material, grain storage, electric power production, and automotive vehicular emissions. Such a concise collection of data can be of considerable usefulness to those working in source apportionment analysis and various aspects of pollution control.

SECTION 2

SAMPLE PREPARATION PROCEDURE

In preparing samples for SEM examination consideration must be given to the type, condition, and data required from the sample. Some data requires that the sample be dispersed well enough to determine particle size and distribution. Depending on the type of sample, e.g., solid bulk, loose powders, or powders on filters, the method of dispersal may require one of the following treatments: ball milling, grinding with mortar and pestle, ultrasonic dispersal, or ashing and redistribution. Obtaining a representative sample is the most significant problem associated with bulk samples. This problem is always addressed in light of the mineral form of the sample. One of the most difficult forms is a mineral like serpentinite where the several forms of the same mineral have different physical characteristics. A preparation procedure addressing this problem has been published (3) and is excerpted below.

Rock fragments should be from 1/2 to 1 in in diameter. About 1/2 pt should be submitted for the analysis. In the laboratory, the sample of quarry rock is placed in a 1-pt roller-type ball mill jar (size 000) and tumbled without balls for 6 to 10 h. Tumbling and mutual grinding of the rocks results in comminution of a layer of material at the rock surfaces and also produces a thoroughly mixed dust sample.

A small sample of the powder (~5 mg) from the ball mill jar is placed in a 50-mm diameter agate mortar. Approximately 0.1 ml of amyl acetate is mixed with the powder in the mortar, and grinding is carried out with a no 3 mullite pestle until the amyl acetate evaporates to dryness (approximately 20 min). The mulling reduces the number and size of asbestos fiber bundles. The mulled sample is transferred to a stainless steel Wig-L-Bug container and mixed for 2 min. (The Wig-L-Bug is a high-speed reciprocating ball in cylinder type grinder, but is used here to mix the powder thoroughly.) A 3-to 10 µg sample is weighed on an electronic balance and placed in a 2-ml glass vial. A total of 100 to 300 µl of 0.001% aerosol OT (as surfactant) is placed in the vial and the suspension is ultrasonicated for 5 min. A Virsonic cell disrupter fitted with a microtip and run at 28% of full power was used in this step, breaking up any remaining fibers or bundles to fibrils.

After ultrasonification the sample is transferred to a 30-ml glass beaker and the volume adjusted to 25 ml by adding 0.001% OT. The sample is covered with parafilm and sonicated for 5 min using a low-powered ultrasonic cleaner.

A 47-mm diameter filtering apparatus is assembled with a 47-mm, 0.1 µm pore size, Nuclepore filter backed up by a 0.8-µm pore size Millipore filter

on the glass frit. Suction is applied and the filters are recentered if necessary. The filter funnel is mounted, the vacuum is turned off, and suction is allowed to cease. The beaker is removed from the ultrasonic cleaner and the aerosol OT suspension of sample is poured into the funnel. The beaker is rinsed 3 times with the solution and the rinsings are added to the suspension in the funnel. Suction is applied and continued until drainage is completed. The vacuum is turned off and the filter is allowed to dry in still air.

After drying, the filter should be carbon-coated immediately (see Section 2.3 in the "Provisional Methodology Manual," EPA Report 600/2-77-178) (4), and analysis for asbestos carried out as described in this manual beginning with Section 2.4. The weight of powdered rock sample and the total deposit area of the filter are taken into account in calculating the fiber mass concentration from the TEM data.

Powders, especially fine powder, can be dispersed by ultrasonification, but some consideration must be given to the dispersing mediums, e.g. whether it reacts with the sample. Generally, amyl acetate is a satisfactory dispersing medium.

Coarse powders either must be ground to smaller size, dispersed in a suitable medium and filtered, or placed directly on a substrate with a small amount of liquid and dispersed by using a wiping action with a needle. Those samples obtained on filters are examined directly, or depending on the filter type, removed by ultrasonification or low temperature ashing. Alternatively, the filter is dissolved and the sample recovered by centrifugation. The particular approach and the required information, establishes the level of care one must take in recovering the sample. Mounting the sample for examination in the SEM also depends on the information required. For a cursory examination of the morphology, the bulk sample can be cemented to an aluminum stub; moreover, elemental data can be obtained from a sample mounted this way provided one realizes that some x-ray scattering from the surroundings can and will be seen in the resulting spectrum.

Fine powders can be mounted by placing a small amount of powder on an aluminum stub, adding a drop or two of amyl acetate, then dispersing it with a probe needle. Where morphology, size distribution, and elemental analysis is needed, the sample must be placed either on a carbon planchet or a beryllium substrate to avoid contribution to the spectra by the substrate, since neither carbon or beryllium x-rays are detected by the spectrometer.

For good size distribution data the sample should be placed in about 5 ml of amyl acetate and ultrasonicated for about 5 min. An aliquote then is placed quickly on a substrate with a pipet. Another approach to good dispersion is to place a small quantity of the sample in distilled water containing a surfactant such as "OT", ultrasonifying for about 5 min, and filtering onto a 0.1 μm Nuclepore filter.

Ambient air samples collected on filters can be examined directly by placing a small piece of the filter onto a carbon planchet or beryllium substrate. However, size distribution is difficult to obtain because of

pooer dispersion, though morphology and chemical composition may be obtained. The chemical composition by x-ray fluorescence will be somewhat unreliable because of the proximity of other particles.

Where size and distribution data are required of the filter sample, a redispersal is necessary. This can be accomplished, if the filter is not glass, by low temperature ashing, redispersing in water-OT by ultrasonification and refiltering onto a 0.1 μm Nuclepore filter, or dissolving the filter with a suitable solvent, centrifuging, rinsing about 3 times in solvent with centrifuging after each rinse, redispersing in water-OT, and refiltering onto a 0.1 μm Nuclepore.

If the sample is on glass or quartz fiber filters, little or no chemical data is obtainable because of the contribution to the spectrum by the various elements in the fiber. Removal of some of the particulate material can be accomplished by placing a piece of the filter in a small beaker in amyl acetate and ultrasonifying. However, for best analytical results, glass or quartz filters should be avoided.

All samples proposed for analysis using the SEM must be coated with an agent that allows the electric charge built up by the electron beam to leak off. Depending on the information needed, these coatings can be single or multiple layers. Where determining chemical composition is important a single layer of carbon should be used. Carbon does not contribute to the x-ray fluorescence spectrum because the x-ray detector is insensitive to x-ray energy of elements with atomic numbers lower than 9.

Where morphology and size distribution are required, a layer of carbon is placed on first, followed by a layer of gold or palladium. In this case, the carbon is used to remove the electric charge while the heavier elements contribute significantly to the secondary electron emission, and thereby, produce higher image contrast and resolution. The coatings required in sample preparation are obtained by vacuum evaporation.

VACUUM-EVAPORATED COATINGS

A vacuum evaporator is usually an apparatus having a set of vacuum pumps, e.g., a mechanical and oil diffusion, and a vacuum chamber such as a glass bell jar. The mechanical pump is used to rough pump the bell jar to about 10 to 50 mm Hg; the diffusion pump is then activated and pumps the chamber to 10^{-5} or 10^{-6} Torr. At that point, a current is sent through a set of electrodes to heat the elements used to coat the sample to the melting point or sublimation point, thereby vapor-depositing a thin coating of the material onto the sample surface. The sample is now ready for examination in the SEM.

Sample preparation for TEM use follows a somewhat different procedure than for SEM use because of sample size restrictions and because of substrate type required.

A sample must be reduced to a powder of 1 μm or less. It can then be placed on a carbon film supported by a 3mm diameter copper grid by first

dispersing the powder in a suitable liquid medium using ultrasonification and then placing a drop of the suspension onto the substrate with a micro pipet.

If the sample is on a polycarbonate filter it can be coated with a carbon film in a vacuum evaporator, and a 3-mm diameter disc cut from the filter. The filter material can then be dissolved away, with a suitable solvent, leaving what is known as an extraction replica containing the particulate material. This procedure, described in detail below, is excerpted from EPA publication 600/2-77-178 entitled "Electron Microscope Measurement of Airborne Asbestos, a Provisional Methodology Manual" (4).

The polycarbonate filter containing the sample deposit and suitable blanks and standards should be coated with carbon as soon after sampling is completed as possible. The carbon coating forms an almost continuous film over the filter and bonds the collected particles to the filter surface. Losses are thus reduced during subsequent handling of the filter, and during the transfer process to the electron microscope grid. A carbon film of about 40 nm thickness is most suitable.

It is highly recommended that the handling and processing of the filters after their receipt by the analyzing laboratory be conducted in a clean room or clean bench to reduce the possibility of contamination. Tweezers should be used for handling the filters; static charge eliminators will facilitate handling of the polycarbonate filters by neutralizing the surface electrostatic charge.

Because a thin, uniform, carbon film is desired, the coating of the filter deposit with carbon should be carried out in a vacuum evaporator. Carbon sputtering devices should be avoided because they produce a film of uneven thickness. Too thick a film can lead to problems during the subsequent steps in the procedure, particularly filter dissolution, fiber sizing, and fiber identification. Electron diffraction patterns tend to be faint when operating the TEM at less than 100 KV.

Typically, vacuum evaporators accept samples as large as 10 cm in diameter. Thus, if the personal sampler was used for sample collection, the entire filter may be carbon-coated at one time. It is convenient to use the petri dish in which the polycarbonate filter is being stored. After inspecting the filter to be sure it is securely tacked to the bottom of the petri dish, the cover is removed and placed in the bottom of the dish containing the filter in the vacuum evaporator for coating. If the airborne asbestos was collected on the 20 cm x 25 cm polycarbonate filter using the high-volume (Hi-vol) sampler, the entire filter cannot be coated at once. Portions, about 2.5 cm x 2.5 cm, should be cut from the central region of the filter using a pair of scissors or a scalpel. The portions should be tacked with cellophane tape to a clean glass microscope slide and placed in the vacuum evaporator for coating.

Any high-vacuum carbon evaporator may be used to carbon-coat the filters (CAUTION: carbon sputtering devices should not be used). Typically, the electrodes are adjusted to a height of 8 to 10 cm from the level of

the turntable upon which the filters are placed. A spectrographically pure carbon electrode sharpened to a 0.1 cm neck is used as the evaporating electrode. The sharpened electrode is placed in its spring-loaded holder so that the neck rests against the flat surface of a second graphite electrode. The samples, in either a petri dish bottom or on a glass slide, are attached to the turntable with double-sided cellophane tape.

The manufacturer's instructions should be followed to obtain a vacuum of about 1×10^{-5} Torr in the bell jar of the evaporator. With the turntable in motion, the carbon neck is evaporated by increasing the electrode current to about 15 A in 10 s, followed by 25-30 s at 20-25 A. If the turntable is not used during carbon evaporation, the particulate matter is not coated from all sides and there is an undesirable shadowing effect. The evaporation should proceed in a series of short bursts until the neck of the electrode is consumed. Continuous prolonged evaporation is not recommended since overheating and consequent polymerization of the polycarbonate filter may easily occur and impede the subsequent step of dissolving the filter. The evaporation process may be observed by viewing the arc through welders goggles. (CAUTION: never look at the arc without appropriate eye protection.) A rough calculation shows that a graphite neck of 5-mm^3 volume, when evaporated over a spherical surface of 10 cm radius, will yield a carbon layer 40 nm thick.

After carbon coating, the vacuum chamber is slowly returned to atmospheric pressure, the filters are removed and placed in clean, marked petri dishes, and stored in a clean bench.

SAMPLE TRANSFER TO ELECTRON MICROSCOPE

The transfer of the collected airborne asbestos from the coated polycarbonate filter to an electron microscope grid is accomplished in a clean room or bench using a Jaffe-washer technique with some modification.

Transfer is made in a clean glass petri dish about 10 cm diameter and 1.5 cm high. A stack of 40 clean, 5 1/2-cm diameter paper filter circles is placed in the dish; alternatively, a 3- x 3- x 0.6-cm piece of polyurethane foam (like those used as packing in Polaroid film boxes) may be used. Spectroscopic grade chloroform is poured into the petri dish until it is level with the top surface of the paper filter stack or the foam. On top of the stack or foam a piece of about 0.6 cm x 0.6 cm 60-mesh stainless steel screen is placed. Several transfers may be completed at one time, and a separate piece of mesh is used for each grid.

Sections of the carbon-coated polycarbonate filter on which the sample is deposited are obtained either by using a punch to punch out 2.3-mm discs or sharp scissors to cut out approximately 1 mm x 2 mm rectangles. A section is laid carbon side down on a 200-mesh carbon-coated TEM grid. (Alternatively, one may use formvar-coated grids or uncoated TEM grids. Here, the carbon coat on the polycarbonate filter forms the grid substrate.) Minor overlap or underlap of the grid by the filter section can be tolerated since only the central 2-mm portion of the grid is scanned in the microscope.

This pair, TEM grid and filter section, is picked up with tweezers and carefully placed on the moist stainless steel mesh of the Jaffe washer. The 1 mm x 2 mm section is wetted immediately by a 5 µl drop of chloroform.

When all the samples are in place in the washer, more chloroform is carefully added to increase the level back to where it just touches the top of the paper filter stack. Raising the chloroform level any higher may float the TEM grid off the mesh or displace the polycarbonate filter section; neither is desirable. The cover is placed on the washer and weighted to improve and seal and reduce the chloroform evaporation.

More chloroform should be added periodically to maintain the level within the washer. After a minimum of 24 h, the polycarbonate filter should be completely dissolved. The TEM grid is removed by picking up the stainless steel mesh with tweezers and placing it on a clean filter. When all traces of chloroform have evaporated, the grid may be lifted from the mesh and examined in the electron microscope or stored for future examination.

SECTION 3

RESULTS AND DISCUSSION

Generally, there are two kinds of filters used in ambient air sampling -- depth-type and screen type. Some membrane filters are sometimes referred to as screen filters even though their structure is neither regular nor defined. They differ from the fiber-depth filter in that they do not contain a random mat of fibers pressed together. The screen type filter is one with pores that penetrate from front to back in a relatively straight line and have openings of uniform size. Micrographs #1 through #10 are some typical examples of these filters.

Micrograph #1. Millipore filter type RAWP. Here the labyrinthian structure is apparent in this membrane filter.

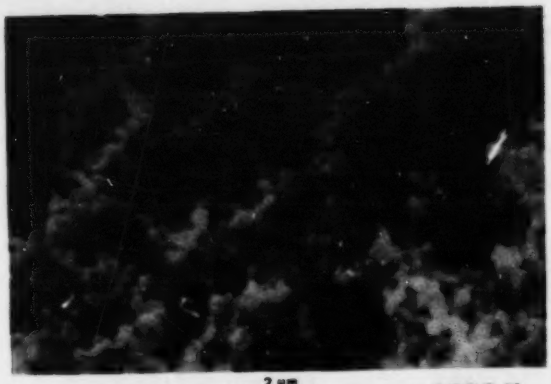
Micrograph #2. Aromatic polymer filter, made by the Gelman Company. It is one of a family of unsupported membrane filters marketed under the name of Metricel. "Unsupported" means that the filters do not include a supporting substrate.

Micrograph #3. Gelman filter marketed under the name of Acropor. These are nylon-supported membrane filters that are used for analytical purposes and can be of hydrophillic, hydrophobic, or ion exchange type.

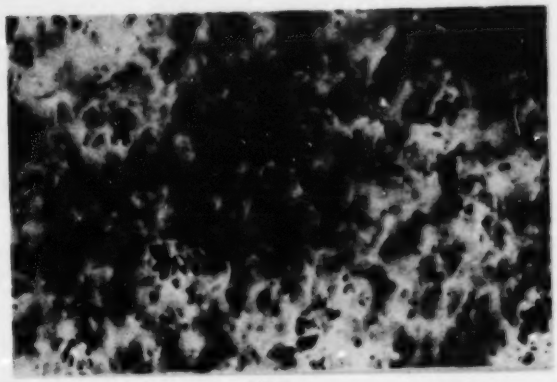
Micrograph #4. Gelman filter called HT Tuffryn of high-temperature membrane type. This filter can be used at dry heat temperatures up to 138°C.

Micrograph #5. Fluoropore filter produced by the Millipore Corporation. The membrane is bonded to a polyethylene net. These filters can be used up to a temperature of 130°C.

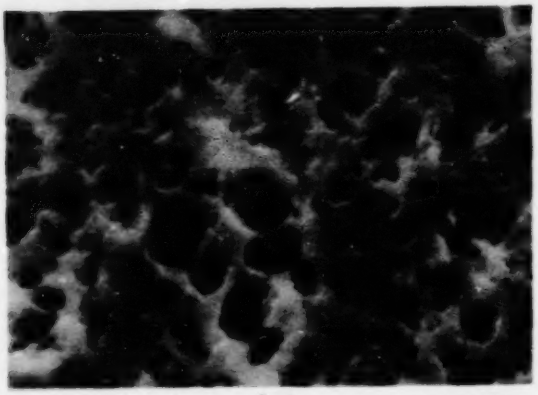
Micrograph #6. Fluoropore filter produced by Millipore Corporation, showing a slightly different structure which results in a larger effective pore size.



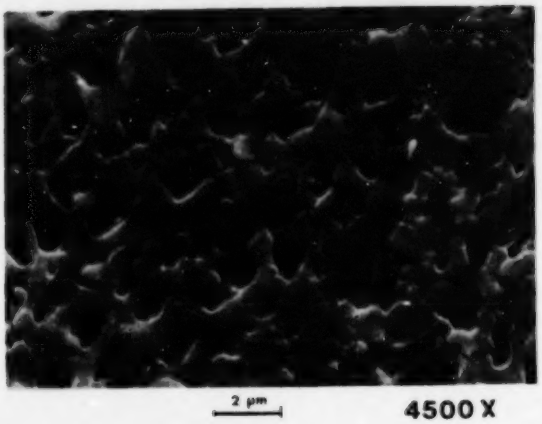
1. Mixed cellulose acetate and nitrate filter



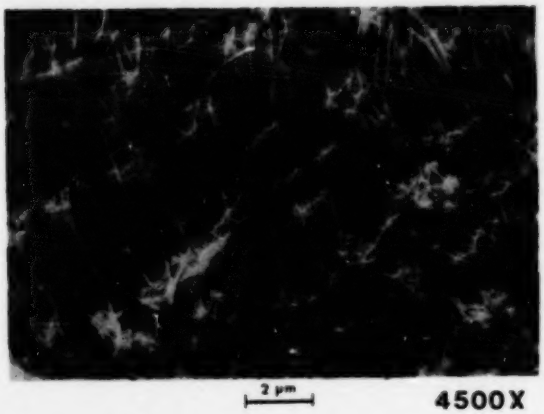
2. Aromatic polymer filter



3. Acrylonitrile polyvinylchloride filter



4. Triacetate filter



5. Polytetrafluorethylene filter

6. Polytetrafluorethylene filter

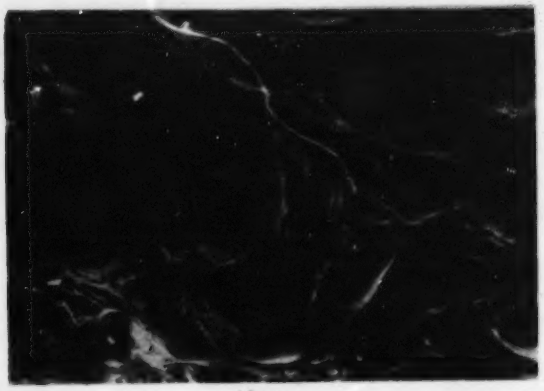
Micrograph #7. Gelman product called Vesapor. It is made of acrylic copolymer with a nonwoven nylon substrate that allows a capacity similar to glass fiber filters.

Micrograph #8. Membrana, Inc., product called Zefluor. It is composed of polytetrafluorethylene, and has no fibrous or net support.

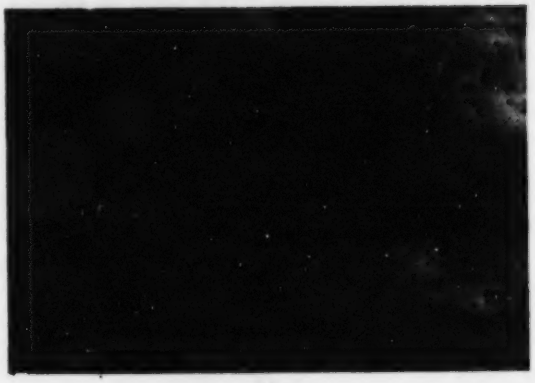
Micrograph #9. Etched-track polycarbonate membrane filter produced by the Nuclepore Corporation. This filter more nearly approaches the two-dimensional screen type than do other membrane filters. Its structure results in a more sieve-like filtration.

Micrograph #10. Typical depth filter fabricated from microfilaments of glass. This filter has wide application in air pollution sampling, but limited application in obtaining samples to be examined by analytical electron microscopy.

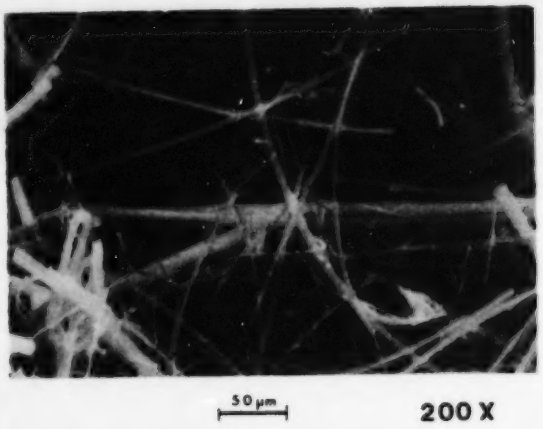
7. Acrylic copolymer filter



8. Polytetrafluorethylene filter



9. Polycarbonate filter



10. Glass filter

Micrograph #11. Crushed serpentinite, taken with a TEM and showing many fibers of chrysotile. Serpentinite is a rock composed mostly of serpentine. Serpentine is the name of a group of minerals -- chrysotile asbestos is the best known -- whose composition is $Mg(Si_2O_5)(OH)_4$. There are three polymorphs of serpentine: antigorite -- a platy variety; lizardite -- a fine-grained and platy variety; and chrysotile -- the fibrous variety. All serpentine rock contains a significant amount of chrysotile. One of the principle uses of serpentinite is the construction of road beds.

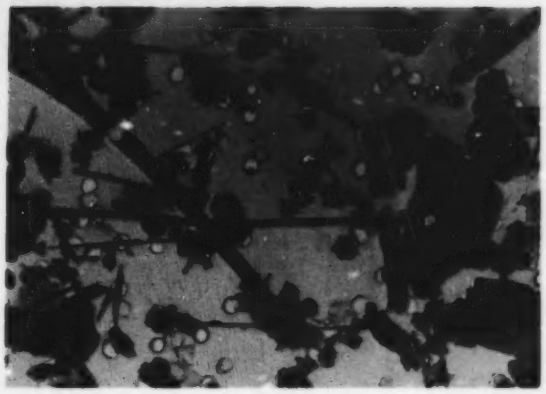
Photograph #12. EDX spectrum showing the chemical composition of bulk serpentinite. The copper is not from the serpentine but from the grid of wires used to hold the specimen in the microscope. The copper spectrum is used to calibrate the x-ray spectrometer.

Micrograph #13. Chrysotile asbestos fibers seen using a SEM and taken from a large solid specimen from a mining operation. Chrysotile occurs as cross-fiber veins varying in size from microscopic to greater than 6 in. Chrysotile is the principle asbestiform mineral used industrially.

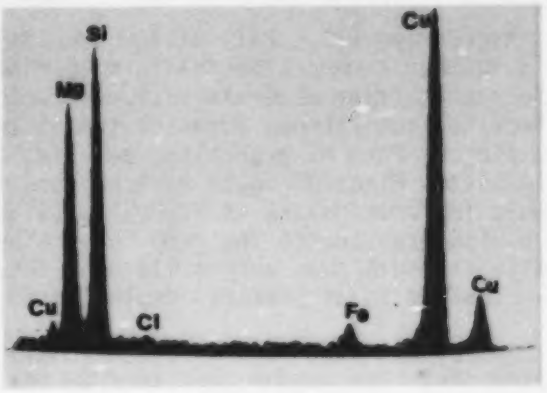
Photograph #14. EDX spectrum showing the chemical composition of bulk chrysotile asbestos. Note the characteristic ratio of magnesium-to-silicon along with some iron, which is usually due to magnetite trapped in the body of the mineral vein.

Micrograph #15. Asbestos insulation debris from building demolition using a SEM to show the morphology of the debris. One of the principle uses of chrysotile asbestos has been as building insulation and as acoustical material. The asbestos was usually bound or combined with other materials as a filler or strengthening agent. In the demolition of old buildings, this material is liberated and falls as debris into the surroundings.

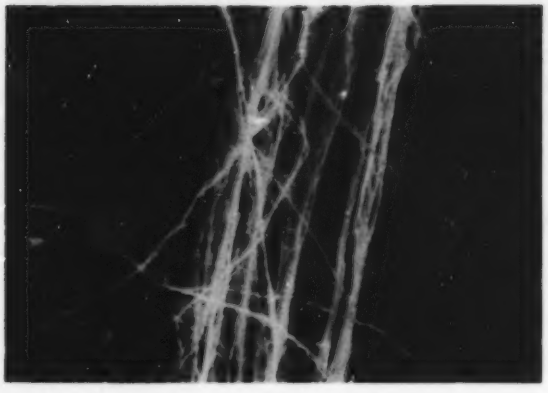
Photograph #16. EDX spectrum showing the chemical composition of asbestos insulation debris.



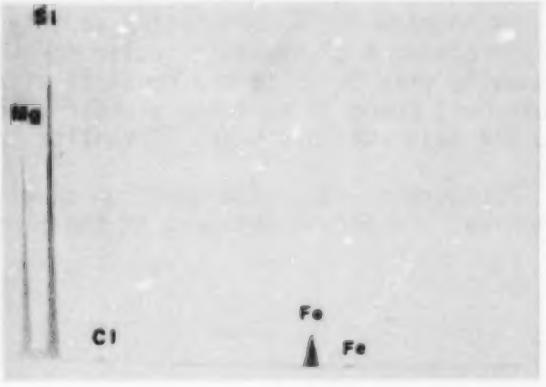
11. Serpentinite



12. Serpentinite spectrum

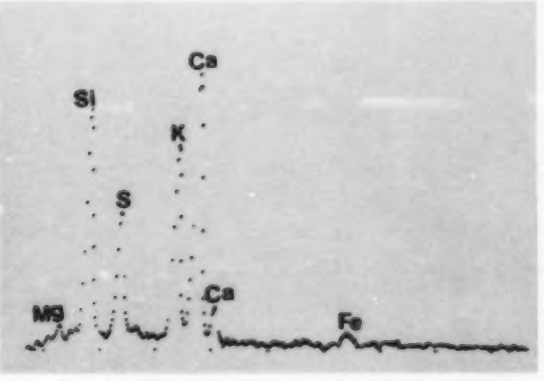


13. Chrysotile asbestos fibers from mining operation



14. Chrysotile spectrum

15. Asbestos insulation debris from building demolition



16. Asbestos insulation debris spectrum

Micrograph #17. Ball-milled crocidolite asbestos taken with a TEM. It is another common asbestos-forming mineral of the amphiboles class. These rock-forming minerals include crocidolite, the fibrous form of riebeckite; the fibrous forms of tremolite, actinolite, grunerite "amosite" (the fibrous form of grunerite, amosite, derives its name from the acronym for Asbestos Mines of South Africa); and anthophyllite. The chemical formula for crocidolite is $Na_6Fe^{2}_{3}Fe^{3}_{2}(Si_8O_{22})(OH)_2$. The fibers are flexible, and bluish gray (hence the name "blue asbestos"), and they have a higher tensile strength than chrysotile asbestos. One of the principle uses for this asbestos is as primary insulation in ship building.

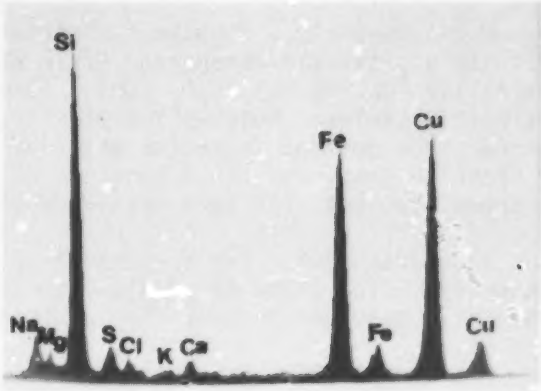
Photograph #18. EDX spectrum showing the chemical composition of crocidolite. The copper peak is from the TEM grid.

Micrographs #19 and #20. Crystal growth habit of crocidolite. The cleavage habit and the cross-parting planes are readily seen.

Micrograph #21. Nonasbestos variety of tremolite taken with a TEM, and exhibiting a bladed-to-acicular morphology. Tremolite ($Ca_2Mg_5Si_8O_{22}(OH)_2$) is usually gray to white and consists of coarse, silky fibers. It occurs most commonly as long, slender needles that radiate in all directions into the adjacent rock body. Tremolite is one of the most common amphiboles.

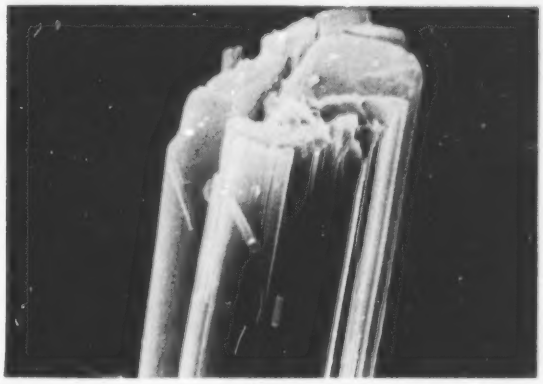
Photograph #22. EDX spectrum showing the chemical composition of tremolite. Copper is not part of the mineral's spectrum.

17. Crocidolite asbestos

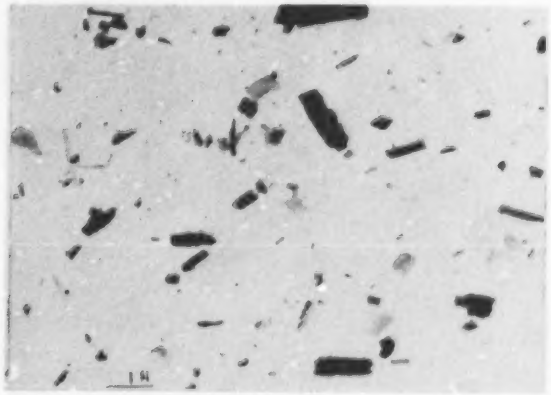


18. Crocidolite spectrum

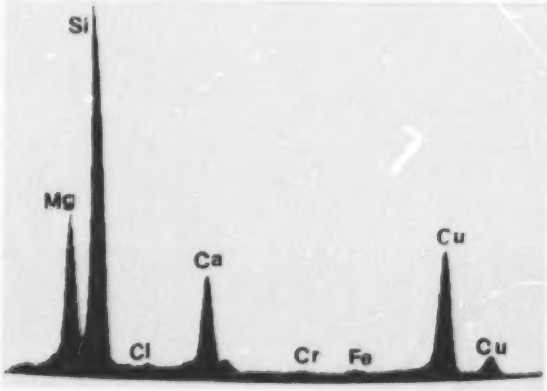
19. Bulk crocidolite



20. Bulk crocidolite



21. Tremolite, nonasbestos variety



22. Tremolite spectrum

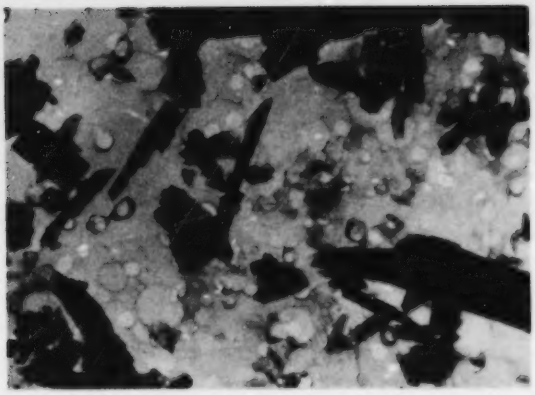
Micrograph #23. Nonasbestos variety of actinolite taken with TEM. It exhibits a prismatic morphology after having been crushed in a ball mill. Actinolite ($Ca_2(Mg,Fe)_5Si_8O_{22}(OH)_2$) is a high iron member of tremolite-actinolite series. However the pure-iron member is not found. Usually the maximum iron content is on the order of 20%. Actinolite is commonly green or greenish gray, and the fibers are quite brittle. Occurrence is similar to tremolite, but with more iron-rich sediments in the vicinity.

Photograph #24. EDX spectrum showing the chemical composition of actinolite. The presence of aluminum indicates that actinolite is compositionally near hornblends. Copper is not part of the mineral's spectrum.

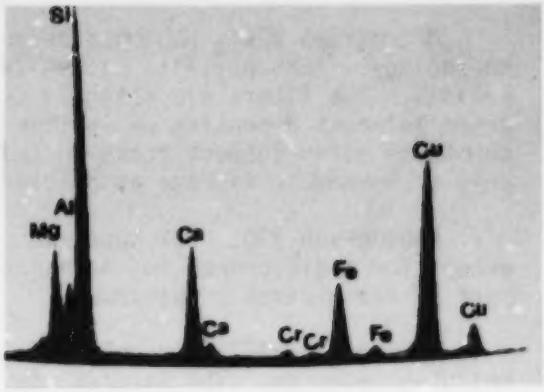
Micrograph #25. Fibrous grunerite or amosite asbestos showing acicular to fibrous morphology and lathe-shaped individual crystal pattern. Amosite asbestos is found in iron-rich sediments that contain little or no sodium. The chemical formula for grunerite (amosite) is $(Mg,Fe)_7Si_8O_{22}(OH)_2$. Amosite used in the asbestos industry is mined in South Africa, but is a contaminant in iron mining operations in the Lake Superior area of the United States. It occurs as cross-fiber veins and is usually brown.

Photograph #26. EDX spectrum of amosite.

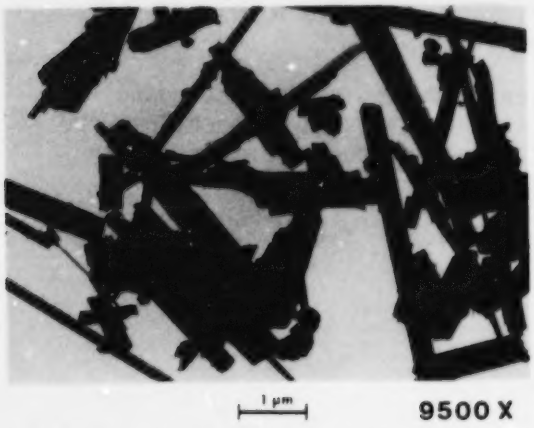
Micrographs #27 and #28. Growth habit of bulk amosite.



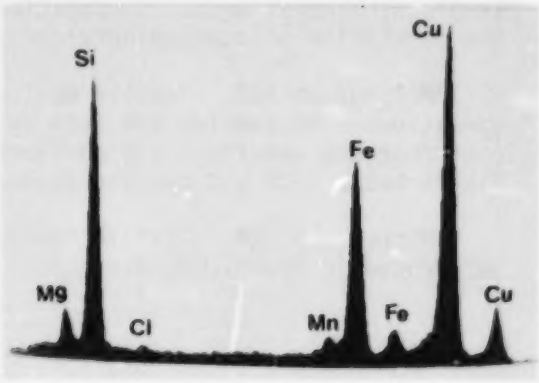
23. Actinolite, nonasbestos variety



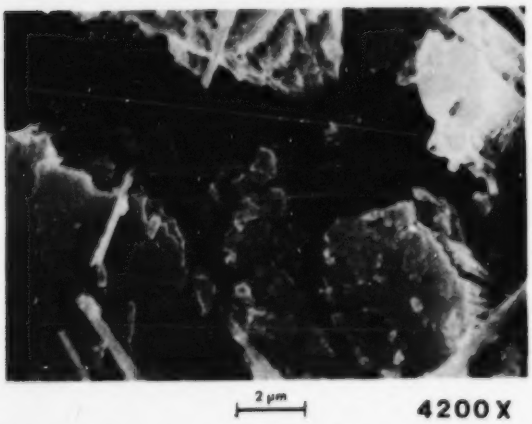
24 Actinolite spectrum



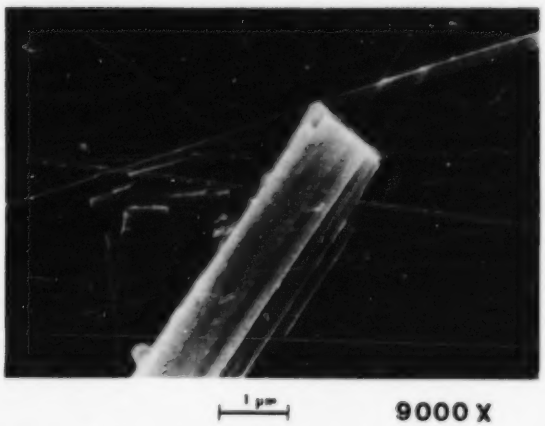
25. Fibrous grunerite, amosite asbestos



26. Amosite spectrum



27. Bulk amosite asbestos



28. Amosite asbestos lathes

Micrograph #29. Nonasbestos anthophyllite showing bladed to acicular morphology. Anthophyllite occurs in short, fibrous cross-fiber veins in schists. The fibers are slightly flexible, and the color varies from green to brown depending on weather exposure. Anthophyllite is fairly common in mafic igneous rocks in the Blue Ridge mountain range. It is the only orthorhombic fibrous amphibole.

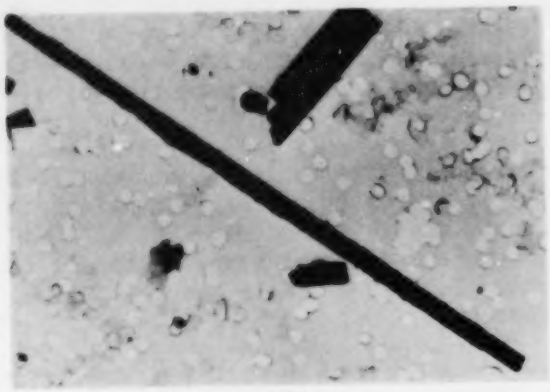
Photograph #30. EDX spectrum clearly resembling that of chrysotile except for a difference in the magnesium-to-silicon ratio. Copper is not part of the mineral's spectrum.

Micrograph #31. Mineral wool, a popular insulating material in the building industry. The material is fabricated from various molten silicates by high-pressure steam or air jet. The fibers seldom reach the small size found in most asbestos.

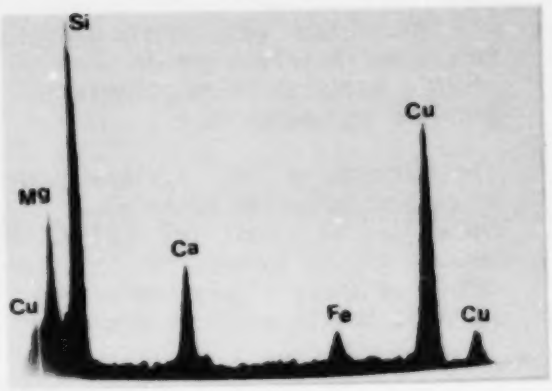
Photograph #32. EDX spectrum showing the chemical composition of this sample of mineral wool. Composition, however, can vary widely depending on the particular silicate mineral or glass being used.

Micrograph #33. Textile dust generated during a cotton carding operation. The samples are from cyclone separations used to collect the dust from the manufacturing environment. This micrograph shows cotton fibers taken from the cyclone separation inlet.

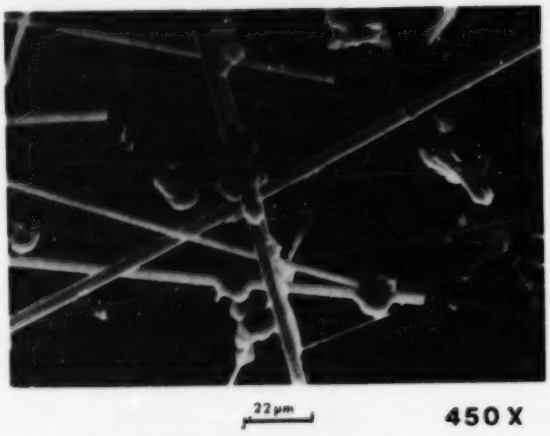
Micrograph #34. Textile manufacturing dust. This respirable dust is implicated in Brown Lung disease.



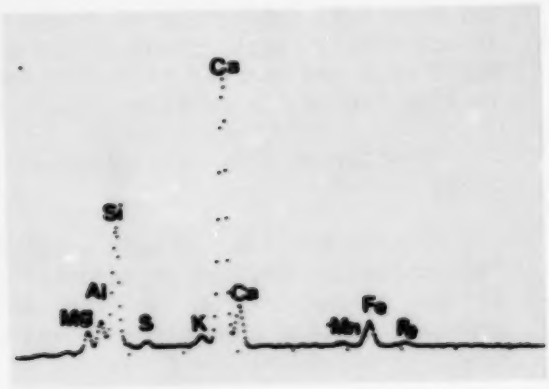
29. Anthophyllite, nonasbestos variety



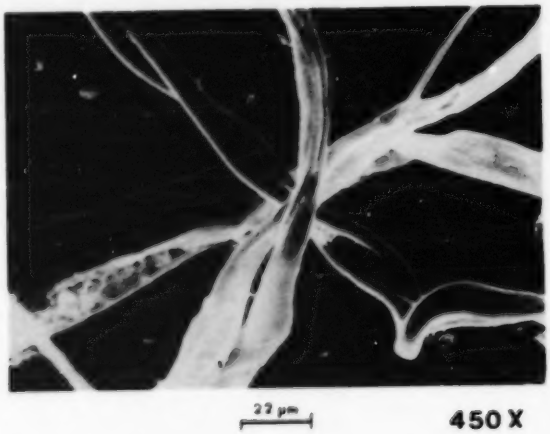
30. Anthophyllite spectrum



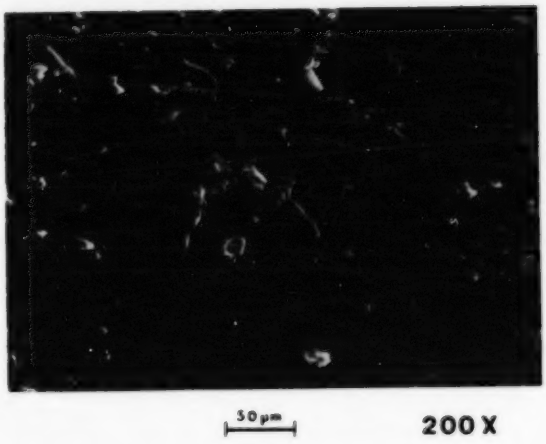
31. Mineral wool



32. Mineral wool spectrum



33. Textile dust



34. Textile manufacturing dust

Micrograph #35. Grain elevator debris. The long fiber like parts are trichomes (hairlike growth from the epidermis of a plant) from wheat. Much of this material is respirable and would tend to cause the same physiological problems as cotton dust.

Micrograph #36. Typical group of graphite fibers used in the manufacture of carbon composite materials. The greatly increased use of carbon-fiber composites in industrial applications could constitute significant hazards because of the susceptibility of electronic and electric power equipment to damage by these highly conducting fibers. Because of their large size potential, health problems are of less concern.

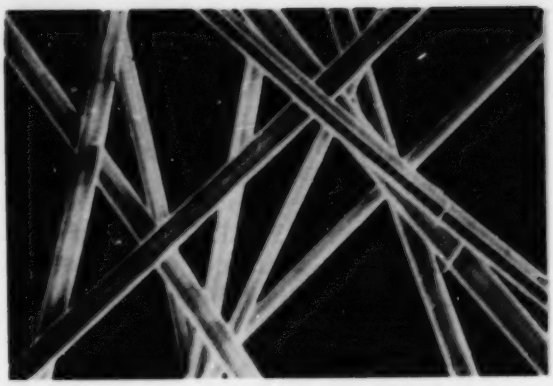
Micrograph #37. Automobile brake drum dust taken with TEM. Brake drum dust consists mostly of iron and iron oxide particles along with mineral dust that has crept in during normal automobile use. However, a small amount of the dust is chrysotile asbestos fibers and fibrils that are small enough to become airborne and respirable. The hazards of respiring asbestos has been well publicized in recent years, and care is now being exercised in automobile repair shops to minimize this danger. The dust shown in this micrograph was treated to remove most of the iron and iron oxide particles, leaving behind the asbestos fibers and fibrils.

Photograph #38. EDX spectrum of the elements found in automobile brake lining.

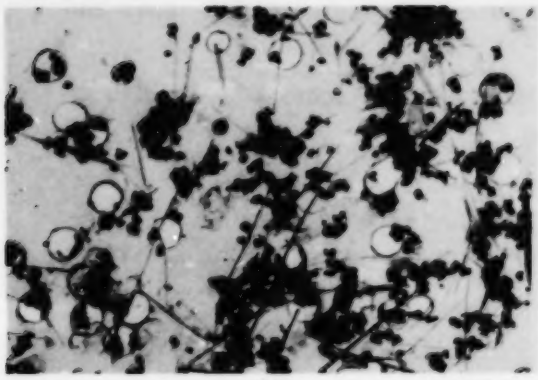
Micrograph #39. Ground automobile exhaust system catalyst material. Anti-pollution devices on automobiles use a catalytic reactor to reduce nitrous oxides, carbon monoxide, and carbon dioxide to a safe level. These reactors contain a material called a catalyst made up of an aluminum silicate material coated with a thin film of platinum.

Photograph #40. EDX spectrum from exhaust system catalyst. The vertical scale of the spectrum has been expanded so that the platinum peak could be seen. This expansion causes the aluminum and silicon peaks to go off-scale.

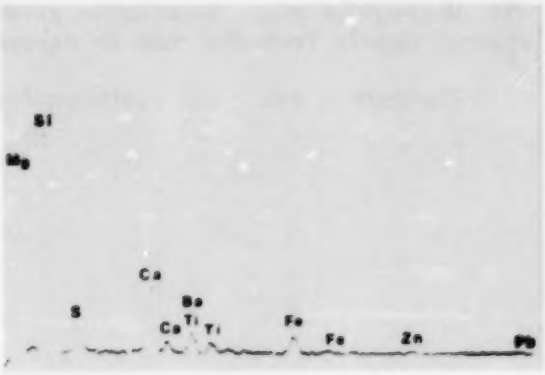
35. Grain elevator debris



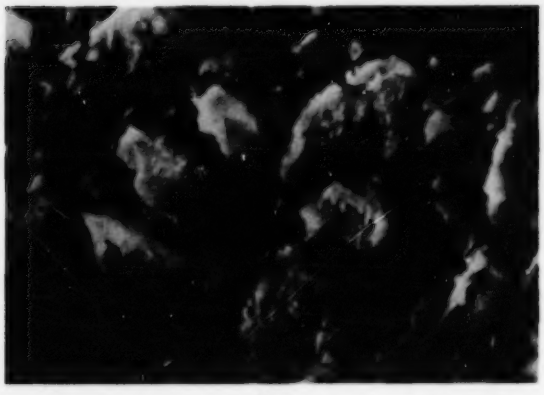
36. Graphite fibers



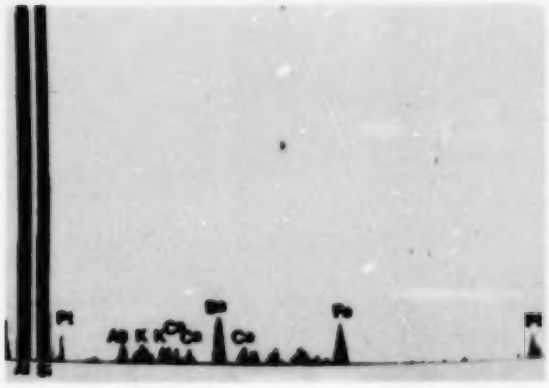
37. Automobile brake drum dust



38. Automobile brake lining spectrum



39. Automobile exhaust system catalyst



40. Automobile exhaust system catalyst spectrum

Micrograph #41. Leaded fuel from automobile exhaust emission. These are agglomerates of necklace-like carbon ribbons on a carbon planchet substrate.

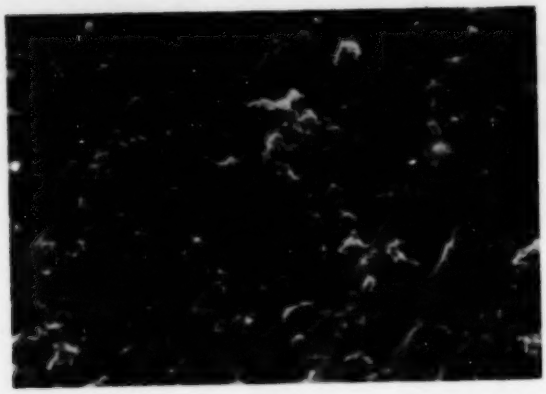
Photograph #42. EDX spectrum from leaded-fuel automobile exhaust emission.

Micrograph #43. Rounded carbon particles from diesel automobile exhaust emission. These rounded particles are characteristic of incomplete fuel burning not only in diesel automobiles but in gas turbines and oil-fired power plants.

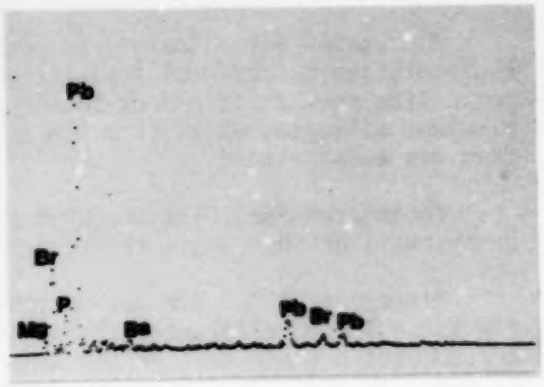
Micrograph #44. Carbon particles from a-25 mega watt electric gas turbine using #2 distillate oil fuel with no additives. The sample was taken from a stack 60 ft from the combustion chamber using a Battelle impact sampler.

Micrograph #45. Automobile exhaust tube sweepings showing sands and mineral debris from the road or pavements.

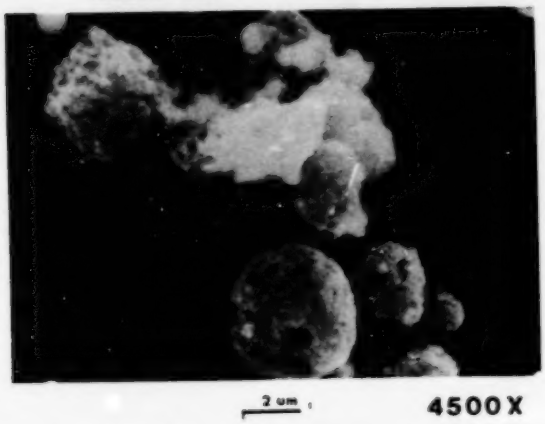
Photograph #46. EDX spectrum from auto exhaust debris.



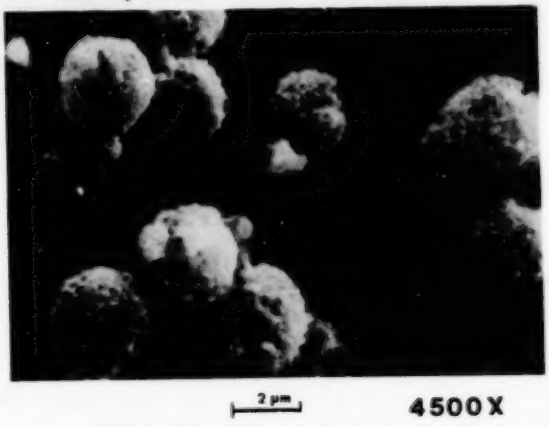
41. Automobile exhaust emission leaded fuel



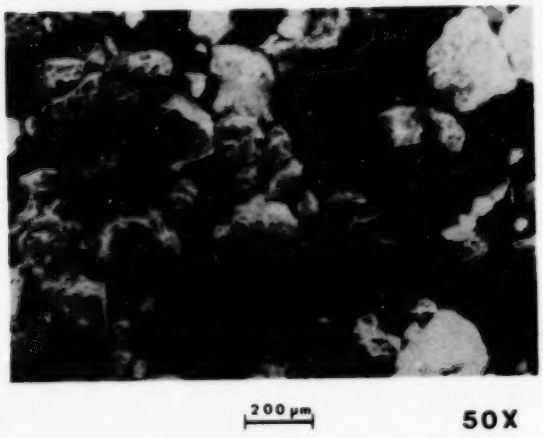
42. Automobile exhaust emission leaded fuel spectrum



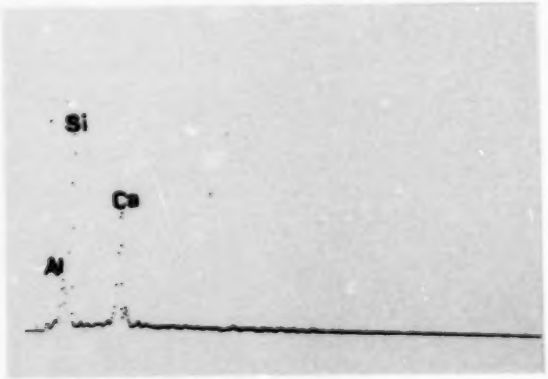
43. Automobile diesel exhaust emission



44. Gas turbine exhaust emission



45. Automobile exhaust tube debris



46. Automobile exhaust tube debris spectrum

Micrograph #47. Coal-fired power plant fly ash showing encapsulation. Encapsulation occurs most frequently when limestone is injected into the combustion chamber to reduce sulfur emission. The shell contains calcium aluminum silicate, which is a lower melting material than the cenospheres that are encapsulated.

Photograph #48. EDX spectrum showing elemental composition of the outer shell of the sphere in micrograph #47.

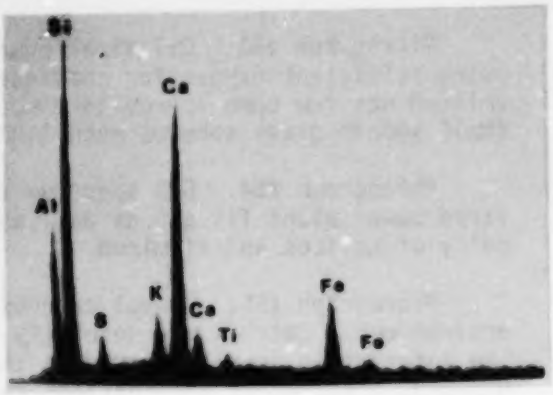
Micrograph #49. Various morphologies found in coal-fired power plant fly ash. The nodular sphere is magnetite. This sample shows a high degree of combustion since most of the spheres are smooth glass.

Photograph #50. EDX spectrum taken of the bulk sample in micrograph #49 showing the high iron peak contributed by the large magnetite particle.

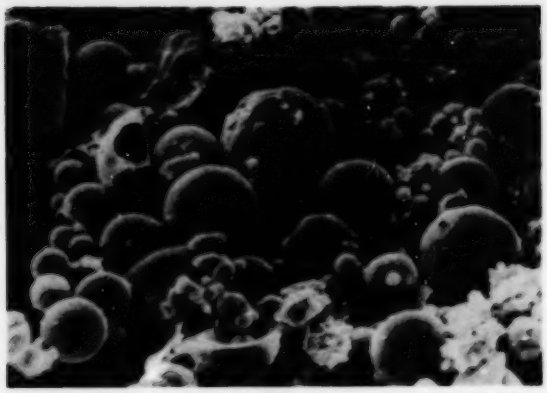
Micrograph #51. Open sponge-like structure of an oil-fired power plant soot particle. The structure results from incomplete combustion. Carbon analysis of these particles showed them to be 30 to 70% carbon. The sample was taken from an operating plant using low oxygen and high vanadium Venezuelan crude oil.

Photograph #52. EDX spectrum from the sponge-like soot particle in micrograph #51. The high background reflects the high carbon content of the particle.

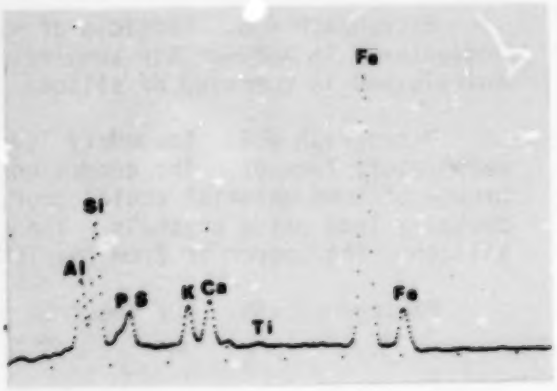
47. Coal fired power plant fly ash showing encapsulation



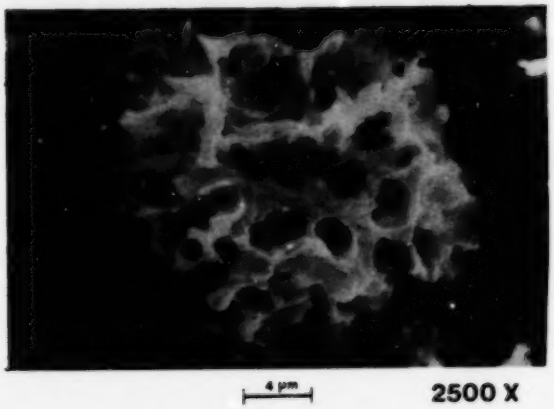
48. Coal fired power plant fly ash spectrum



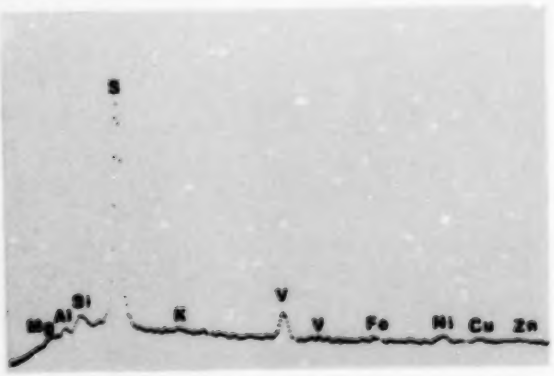
49. Coal fired power plant fly ash showing various morphology



50. Coal fired power plant fly ash spectrum



51 Oil fired power plant fly ash



52. Oil fired power plant fly ash spectrum

Micrograph #53. Oil-fired power plant fly ash. In operating plants using sufficient oxygen for complete combustion of the fuel oil, the fly ash emitted has few open sponge-like soot particles. Instead, the ash becomes small smooth glass spheres much like that found in coal-fired power plants.

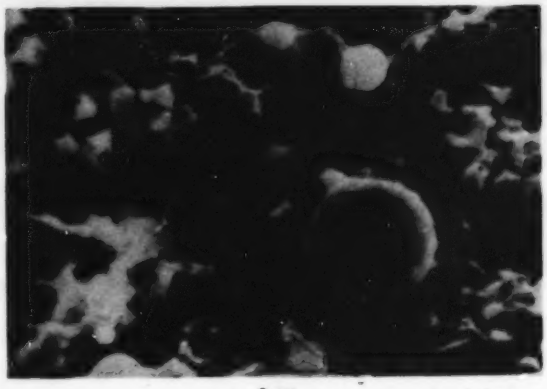
Photograph #54. EDX spectrum reflecting complete combustion of oil-fired power plant fly ash as well as reduction of the ash to a glass principally of silicon and aluminum.

Micrograph #55. Hi-vol catches of ambient air taken from an urban environment. Catches are generally made on large glass fiber filters because the information usually needed is the weight of material suspended in the air. However, microscopic examinations can be and are made from these catches, and they do reflect the contributions of the surrounding environment. Urban, manufacturing districts, rural, proximity to highways, power plants, etc. all contribute a somewhat distinguishing morphology to the overall catch.

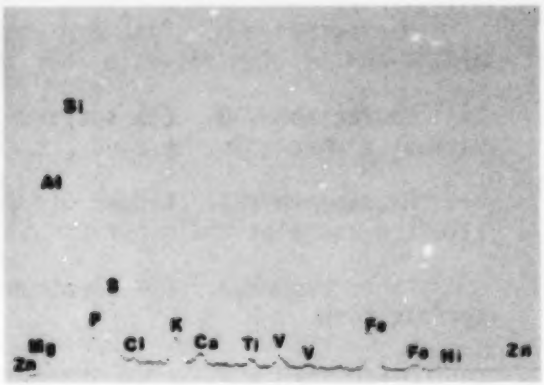
Micrograph #56. Particle of diatomaceous earth, a material frequently encountered in ambient air samples. It is the skeletal remains of tiny marine animals and is composed of silica.

Micrograph #57. Secondary lead smelter emissions taken from a reverberatory furnace. The conditions in the furnace were such that terminal growth of some material could occur, forming rhombic crystals; they are most probably lead oxide crystals. The gray spongy material contains aluminum and silicon. The copper is from the TEM specimen grid.

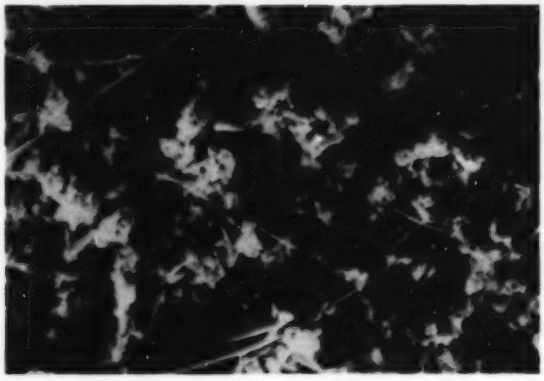
Photograph #58. EDX spectrum of secondary lead smelter emissions.



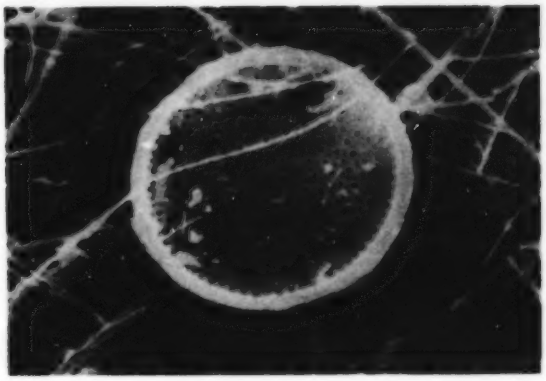
53. Oil fired power plant fly ash



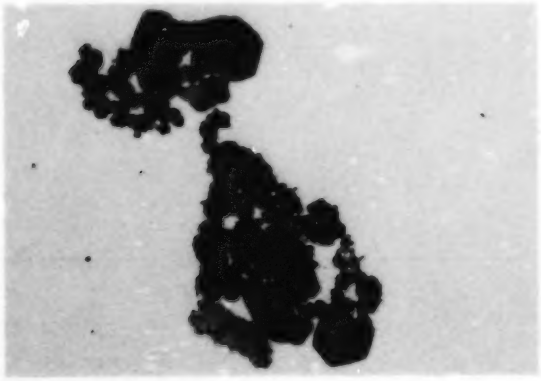
54. Oil fired power plant fly ash spectrum



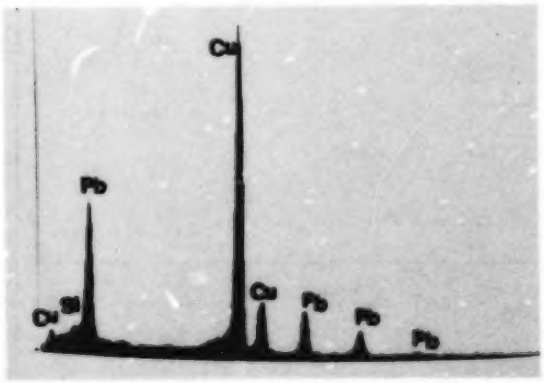
55. Ambient Hi-vol catch



56. Diatomaceous earth



57. Secondary lead smelter emissions



58. Secondary lead smelter emissions spectrum

Micrograph #59. Zinc ore concentrate. Zinc ore contains some iron pyrite and calcium silicate, but is mostly β-zinc sulfide.

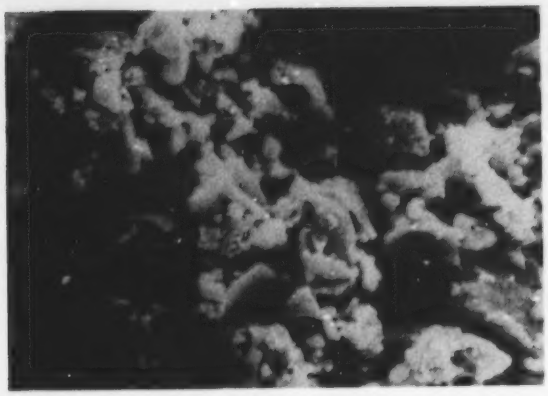
Photograph #60. EDX spectrum showing the relative abundance of the various elements in the zinc ore concentrate.

Micrograph #61. Copper ore concentrate. Principally chalcocite (Cu_2S), it often contains chalcopyrite which contains iron ($CuFeS_2$).

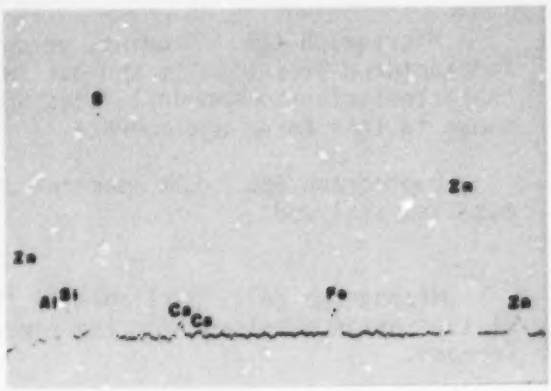
Photograph #62. EDX spectrum of copper ore showing the presence of the pyritic form.

Micrograph #63. Lead ore concentrate. The principle mineral of lead is Galena which is usually associated with zinc ores. Good cleavage is a quality of Galena that makes it easily recognized. Some of this cleavage is seen in particles in this micrograph.

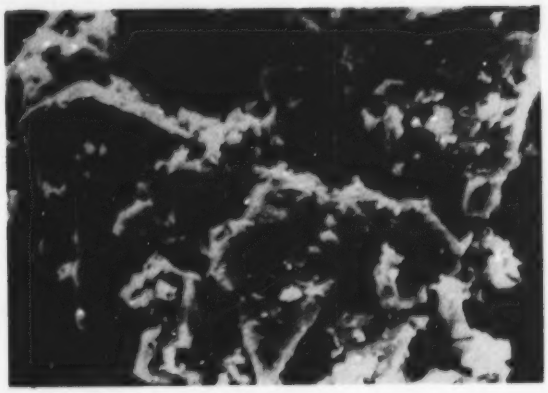
Photograph #64. EDX spectrum from the lead ore concentrate showing the presence of zinc.



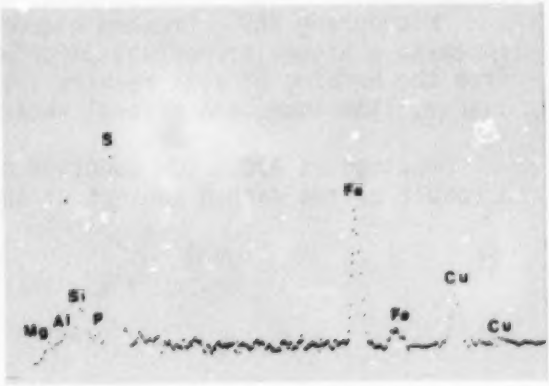
59. Zinc smelter particulates



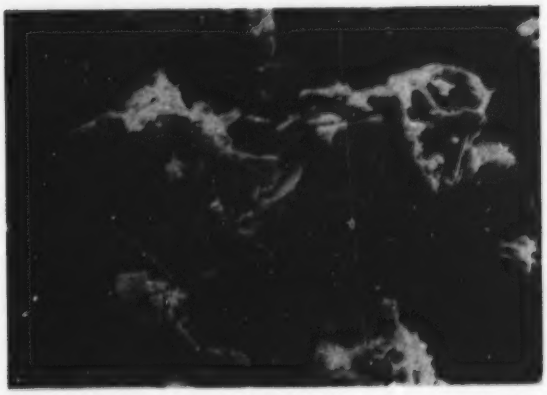
60. Zinc smelter particulates spectrum



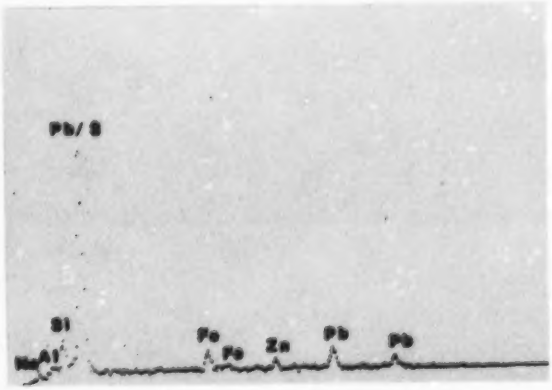
61. Copper ore concentrate



62. Copper ore spectrum



63. Lead smelter concentrate



64. Lead smelter concentrate spectrum

Micrograph #65. Alumina, or corundum in the natural state. Alumina is manufactured from bauxite and has replaced corundum as an abrasive. The characteristic rhombohedral faces and barrel-shaped hexagonal pyramids are shown in this fused agglomerate.

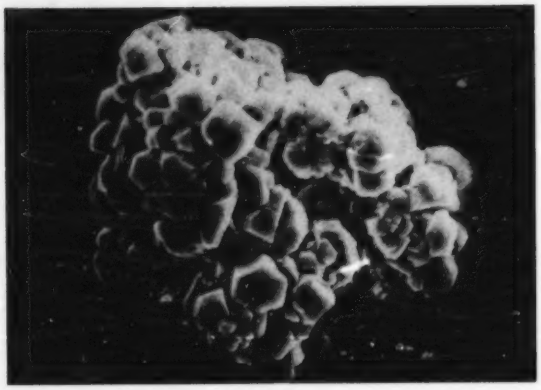
Photograph #66. EDX spectrum of alumina reflecting the purity of the material analyzed.

Micrograph #67. Particulates from steel mill showing submicron spheres of iron oxide obtained from the electrostatic precipitator of a basic oxygen furnace.

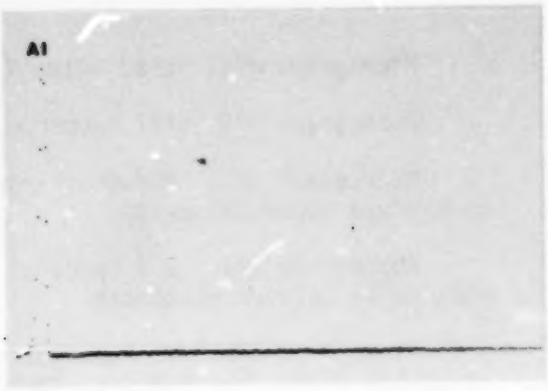
Photograph #68. EDX spectrum from steel mill particulates. The silicon and potassium are from slag material that generally is present in such emissions.

Micrograph #69. Crushed bituminous coal. This is an eastern coal and contains a higher percentage of potassium than western coals. The fly ash from the burning of coal results from such impurities as shale, clay, slate, quartz, limestone, and mineral residue of plant life.

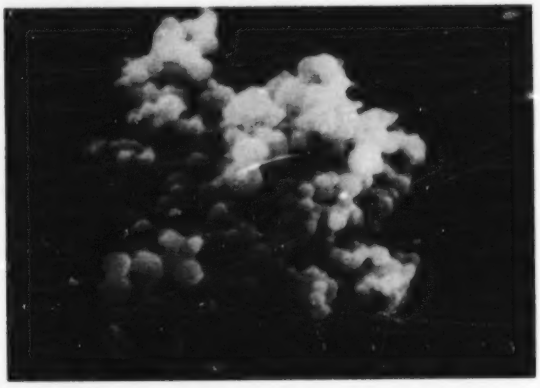
Photograph #70. EDX spectrum of crushed coal. The high background is a result of the carbon content of the coal.



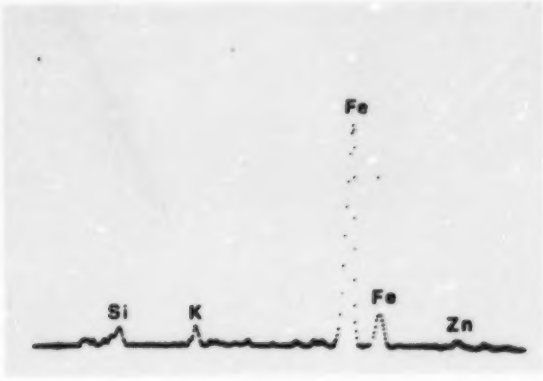
65. Alumina particle



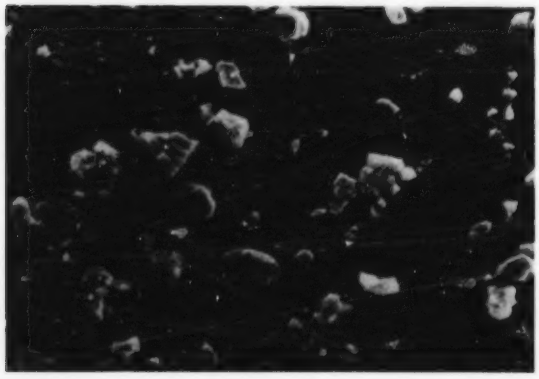
66. Alumina particle spectrum



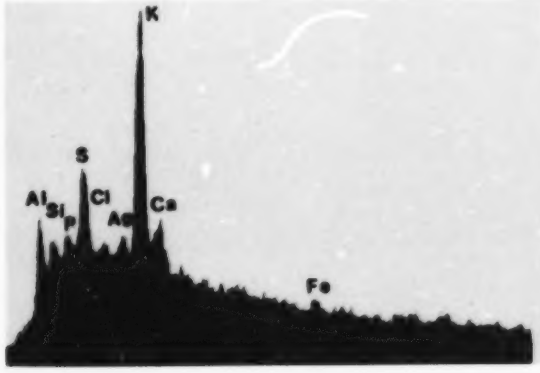
67. Particulates from steel mill



68. Particulates from steel mill spectrum



69. Crushed coal



70. Crushed coal spectrum

Micrograph #71. Lead oxide spheres taken from a lead smelter baghouse.

Photograph #72. EDX spectrum of lead smelter baghouse spheres.

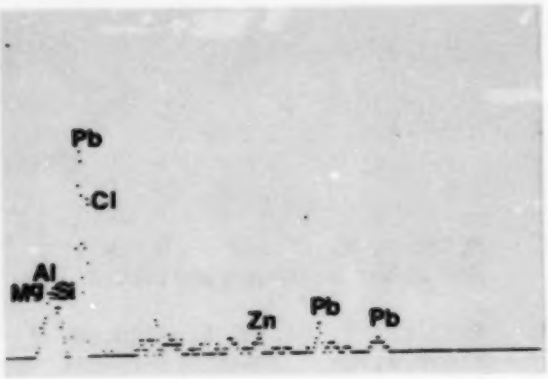
Micrograph #73. Phosphate rock feed used in the fertilizer industry to produce super phosphates.

Photograph #74. EDX spectrum from the phosphate rock feed showing the rock to be calcium phosphate.

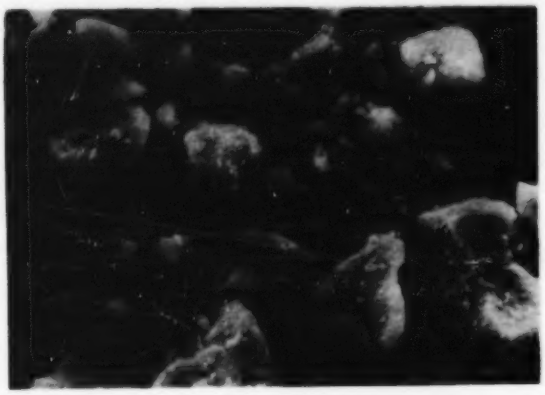
Micrograph #75. Mt. St. Helen's ejecta particulates. During the first eruptions of Mt. St. Helen's, large quantities of ash were ejected into the atmosphere. Here the morphology of this ejecta is shown to be a frothy glass, with sharp fracture edges.

Photograph #76. EDX spectrum of Mt. St. Helen's ejecta showing principally a calcium aluminum silicate, very rich in silica.

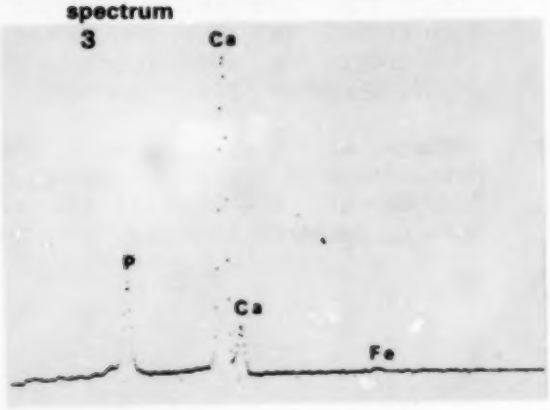
71. Lead smelter baghouse particulates



72. Lead smelter baghouse particulates spectrum

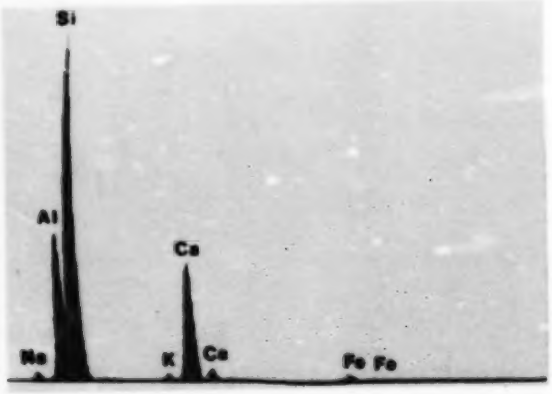


73. Phosphate rock feed



74. Phosphate rock feed spectrum

75. Mt. St. Helen's ejecta particulates



76. Mt. St. Helen's ejecta particulates spectrum

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